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In: Performance of Protective Clothing, v. 4; ASTM STP 1133 (1992)

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REFERENCE: Laughlin, J.M. **"Statistical Fit Models of Methyl Parathion Decontamination from Applicator Clothing,"** Performance of Protective Clothing: Fourth Volume, ASTM STP 1133, James P. McBriarty and Norman W. Henry, Eds. American Society for Testing and Materials, Philadelphia, 1992.

ABSTRACT: Temperature of washing, detergent type, detergent concentration, prewash product use, and level of water hardness were evaluated for completeness of methyl parathion residue removal from unfinished and repellent finished cotton/polyester fabrics during laundering. The objective was testing of models of detergent type, concentration and mineral content of water with temperature of refurbishment. As level of water hardness increased, residue removal was less complete; however, use of a prewash product was effective in off-setting the decreased cleaning efficiency. Refurbishment was more effective at the elevated temperature, but detergent concentration was seen to compensate for lower refurbishment temperature.

KEY WORDS: Pesticide, pesticide residue, protective clothing, laundering, functional finish.

Appropriate laundering conditions can reduce pesticide residues in fabrics [1]. Elevating washing temperature [2, 3] using a heavy duty liquid detergent [2] or phosphate detergent [4], applying prewash product [5], including a prerinse in the laundering procedure [3, 6], and washing clothing daily [7] have been found to reduce pesticide residues in fabrics. Even with vigorous laundering, occurrences of organophosphate poisoning traced to persistence of pesticide in protective clothing continue to be reported [8].

The model for removal of soil from fabrics is proposed as appropriate for decontamination of textiles. Successful refurbishment of textiles is contingent upon the three interacting forms of energy: chemical, thermal and mechanical. If one of these energies is diminished, the outcomes of the laundering process might be maintained at an optimal level through increasing one of the other energy factors. For example, water with more than 150 ppm mineral content can affect the available chemical energy of detergents in the refurbishment process. When an anionic detergent is used, the anionic surfactants ionize and the negative charge of the surfactant attracts the positively charged ions in water. This

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interaction reduces the effectiveness of the detergent. Nonionic surfactants are insensitive to levels of water hardness.

Laughlin and Gold [9] reported that methyl parathion residue in fabric is affected by water hardness and combined factors of detergent type and use of prewash product. Residue removal was lower in repellent finished fabrics, and greater residues were found when anionic phosphate detergents were used without the companion use of a prewash product. However, the work did not address whether increasing the concentration of detergent could provide similar compensation, and whether temperature of washing (thermal energy) changed the residue remaining after laundering.

METHODS

The purpose of this study was to develop and test statistical models of chemical energies interacting with thermal energies. The models under evaluation included the effect of levels of water hardness, detergent type, detergent concentration, use of prewash product or temperature of washing. The factorial experiment design was reported in earlier work [10].

Fabric Specimens

The fabric was an unfinished bleached and mercerized 50% polyester/50% cotton poplin. The fabric was evaluated unfinished (UN) and soil repellent finished (SR) with 1.0 percent (w/w) add-on of a renewable fluorocarbon aerosol. Specimens, 8 x 8 cm, were randomly selected from prepared fabric. The outer 10 percent of the yardage was removed prior to preparation of test specimens as described in the ASTM Test for Breaking Load and Elongation of Textile Fabrics [D 1682-64 (1975 R-82)] to ensure consistency of fabric parameters under evaluation.

Contaminating and Laundering the Specimens

Methyl parathion (MeP) (0,0-dimethyl 0-p nitrophenyl phosphorothioate) dilutions were prepared at 1.25 percent AI (active ingredient) field strength concentration from an emulsifiable concentrate formulation (54% AI) as reported in earlier work [10]. Two hundred µL were pipetted onto the center of a specimen. Contaminated specimens were air dried four to six hours.

Following contamination and drying, specimens were laundered and analyzed, or were analyzed unlaundered (controls). Water for each level of hardness was made up from stock solution as per ASTM D 4008-81, 7.6 Test Method for Measuring Anti-Soil Deposition Properties of Laundry Detergents following the method of Easter and Lietzke [11]. That is, the 1000 ppm water was prepared by dissolving 14.7 g $CaCl_2 \cdot 2H_2O$ and 10.165g $Mg\ Cl_2 \cdot 6H_2O$ in 1,500 mL distilled H_2O at 7.0 $\pm$ 0.2 pH and at twice these concentrations in 1,500 mL H_2O for 2,000 ppm hardness level. The water used for the research was at 0, 1,000 and 2,000 ppm mineral content. Specimens were laundered in a LaunderOmeter (Model LEF) using a modified AATCC (61-1975) Test for Colorfastness to Washing, Domestic, and Laundered, Commerical: Accelerated [12] with a nonionic/anionic heavy duty liquid (HDL) detergent or a built 8.6% phosphate (PHOS) detergent. The wash liquors were made up at concentrations of 0.0*x* (no detergent), 1.0*x*, 1.5*x* and 2.0*x*, with *x* being the concentration recommended by the detergent manufacturer on the package label. The HDL protocol was 4 mL/996 mL water, 6 mL/994 mL water and 8 mL/992 mL water and the PHOS protocol was 2.17 g/998.83 mL water, 3.25

g/996.75 mL water and 4.33 g/995.67 mL water. Refurbishment was done at 30°C or 60°C. One of each paired set of specimens was pretreated with a prewash product applied at a volume of 0.250 mL immediately prior to laundering. All work was replicated three times.

Residue Analysis

MeP was hexane extracted from fabric specimens; then analyzed on a Varian Vista 3400 gas chromatograph with electron capture detector, as reported in earlier paper [10]. Separation was achieved on a 2 m x 2 mm glass column packed with 3 percent OV-101 on 80/100 mesh Chromosorb W-HP with nitrogen flow of 40 mL/min. Injection, detector, and oven temperature were 285°C, 325°C, and 220°C, respectively. Total amount of MeP residue in each specimen was expressed in $\mu g/cm^2$. Baseline aliquots for determining recovery rate were subjected to the same analysis. Recovery rate of 99% was used in calculating after-treatment residues.

Statistical Analysis of Data

The purpose of this study was to test models of combinations of thermal and chemical energies; therefore the statistical analysis reported here addressed model testing. The statistical analyses of data were based on amount of pesticide residue remaining in the fabric after treatment and the proportion of the initial contamination as after-laundering residue (percent residue remaining). Since these percentages were not normally distributed, arcsine transformations (arcsine $\sqrt{Y}$) were applied to the percentages to assure that the data conformed to assumptions underlying ANOVA. Following General Linear Model Analysis, orthogonal contrasts were used to establish linear, quadratic, and cubic relationships. Comparisons of parametric estimates between linear models were performed. The decision level was $p \leq 0.05$.

Phase I - Unfinished Fabrics

Laundering significantly decreased the amount of MeP on the fabric specimens. Main effects attributable to temperature of washing, to detergent concentration, and to prewash product use were revealed along with interaction effects of detergent type *x* temperature of washing *x* detergent concentration [10].

Use of prewash product enhanced residue removal, with 5.55% initial contamination when the prewash product had been used in comparison to MeP residues of 12.99% of initial contamination when not used. It is important to reiterate that there was an interaction of the factors of detergent type, temperature and detergent concentration; that interaction must be studied before impact of these main effects can be established. To elucidate this interaction, a graph (Figure 1) was prepared and orthogonal contrasts used to determine if the relationships among temperature and detergent type across detergent concentrations was linear in nature. The polygonal contrasts confirmed a linear effect for detergent concentration when amount of residue was the response variable ($F=3.49$, $p \leq 0.05$) and when percent residue was the response variable ($F=4.98$, $p \leq 0.05$) as well as a cubic effect for detergent concentration when amount of residue was the response variable ($F=4.65$, $p \leq 0.05$).

Post hoc orthogonal comparisons were used in an attempt to partition this cubic model for interacting factors. A significant linear interaction was found for temperature with detergent type for each detergent concentration ($F=5.23$, $p \leq 0.05$), but comparisons were not significant when interactions of prewash with detergent concentration (linear, quadratic, cubic) were considered.

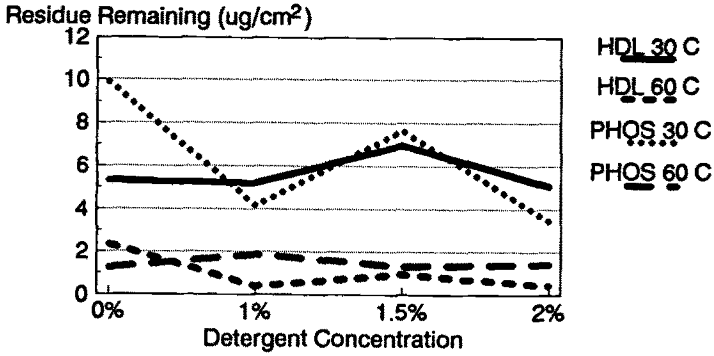

FIG. 1--Interaction of detergent type, temperature and detergent concentration on MeP residue remaining on unfinished fabrics. (Includes data for prewash product use and for laundering without a prewash product.)

Visual inspection of the graph of the residues (Figure 1) was as enlightening as the partitioning for orthogonal contrasts. Given that the 0% concentration of detergent included both those specimens laundered with and without a dosage of prewash product (for which a main effect was found), it was necessary to examine this more closely. The prewash product contained surfactant, but no surfactant (detergent or prewash) was present when laundering was done without detergent and without prewash product. That is, laundering was done with water only.

The residues remaining after laundering were affected by the temperature at which laundering was done. Two levels of responses were noted, with less than 2.0 $\mu g/cm^2$ for laundering with prewash product and no detergent at 60°C and more than 5.0 $\mu g/cm^2$ for laundering at 30°C.

A *post hoc* analysis of the four regression models in Figure 1 was completed following the visual inspection. Using PROC REG in SAS, the four models (detergent type and temperature across detergent concentrations) were evaluated to determine whether the four slopes were "the same" (Ho: $\beta_1=\beta_2=\beta_3=\beta_4$). The resultant F value of 1.9135 (d.f.=3,8) was not significant, therefore the null hypothesis stood and the four slopes were essentially identical, even though at different placements on the *y* axis. To confirm this placement on the *y* axis, the intercepts were evaluated to establish whether they were "the same" (Ho: $\alpha_1=\alpha_2=\alpha_3=\alpha_4$). This analysis resulted in a decision to reject the null hypothesis (F=8.05, d.f.=3,8, p=0.008) and to conclude that there was not a common intercept"°."

When the four models were tested to establish whether they were identical (Ho: $\alpha_1=\alpha_2=\alpha_3=\alpha_4$ and $\beta_1=\beta_2=\beta_3=\beta_4$), the analysis (F=8.59, d.f.=6,8, p=0.003) resulted in a decision to reject the null hypothesis, and conclude the four models were not identical due to the intercept placement. Temperature and detergent type produce dissimilar results across detergent concentrations. HDL at 60°C was different from PHOS at 30°C (F=16.11, d.f.=1,8, p=0.004) and PHOS at 60°C was different from PHOS at 30°C (F=19.01, d.f.=1,8, p=0.002).

LS Means contrasting the points on each model were used to establish where the significant differences among means occurred in the models. The 0.0x concentration results were not significantly different at 60°C and 30°C. There was no difference between the HDL and PHOS laundering done at 30°C regardless of detergent concentration. Residues remaining were different between the two temperatures at each concentration when either PHOS or HDL had been used at 60°C, but each concentration of detergent was not different from the other concentrations for each detergent used at 60°C. Washing temperature had a greater impact than detergent concentration in completeness of refurbishment relative to residues remaining.

Repellent Finished Fabrics

Residue reduction was not as large in the repellent finished (SR) specimens as it had been in the unfinished (UN) specimens, even though the initial contamination of the SR had been less than the UN specimens. The MeP on the SR specimens was 7.34 µg/cm^2 compared to residue of 3.55 µg/cm^2 MeP on UN. Removal of MeP from SR finished fabrics is problematic. Current recommendations are for SR finishes to decrease the fabric absorption of chemical. A parallel concern is to lower post-laundering residues of pesticides to the smallest amount possible.

When the General Linear Analysis was employed, main effects were found for detergent type, temperature and use of prewash product. Interaction effects were found for detergent type with prewash product, for detergent type with detergent concentration, and a three-way interaction of detergent type, temperature and prewash product use. Since the three-way interaction and the other two interactions contributed to each main effect, these interactions were plotted (Figure 2).

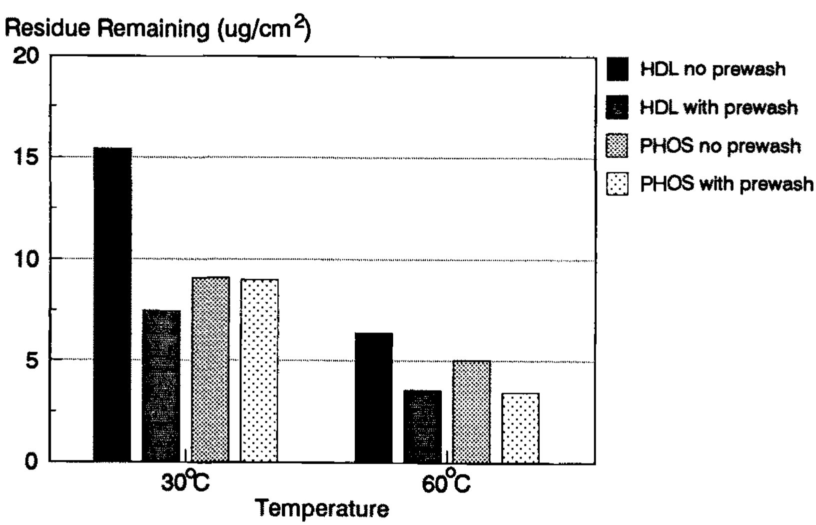

FIG. 2--Interaction of detergent type, temperature and prewash product use on residue remaining in laundered repellent finished specimens.

Comparison of Models

Laundering significantly decreased the amount of MeP on the fabric specimens. Residue levels were greater for the SR finished fabric than for the UN fabric, even though the initial contamination of the SR fabric specimens had been less than half that of the UN fabric specimens [10]. Therefore these data were subjected to concatenated General Linear Model Analysis. Statistically significant differences in amount of residue remaining in specimens after laundering were found attributable to main effects of fabric finish as well as temperature and use of prewash product. Interactions were observed for detergent type with finish, for detergent type with detergent concentration, for finish with detergent concentration, and three-way interactions of detergent type *x* prewash *x* finish and detergent type *x* temperature *x* detergent concentration.

The four-way interaction of detergent type *x* temperature *x* prewash *x* finish was studied. As illustrated in Figure 3, the most successful results (lowest residue remaining after laundering) were achieved in the two instances when the unfinished fabric had been laundered at the higher temperature (60°C) with the use of prewash product to assist either detergent. The lower temperature, 30°C, resulted in greater residues on the SR specimens. Use of prewash product generally assisted in lowering residues, but the contribution was not as marked on the SR specimens laundered in the PHOS detergent at 30°C. In all other instances, use of a prewash product decreased residues to approximately half of those found when a prewash product had not been used.

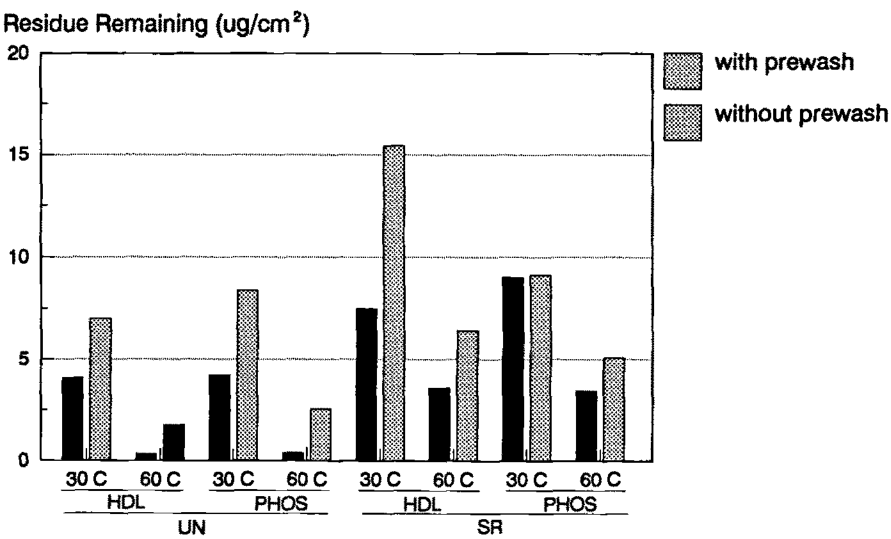

FIG. 3--Interaction of detergent type, temperature, prewash product use and fabric finish on amount of MeP residue remaining after laundering.

Procedures for determining whether the detergent type, temperature and prewash product use performed in similar ways across the increasing levels of detergent concentration included visual inspection of the models, or using LS means to compare points on the models, or employing statistical testing of the models. Statistical model analysis was used to test the comparability of successive levels of detergent concentration for each detergent type/temperature combination on the amount of residues retained by the specimens. Using PROC REG in SAS for testing the statistical fit of the models (Figure 4) these were determined not to be identical ($F=22.21$, d.f.=6,8, $p \leq 0.01$). Visual inspection indicated models with similar intercepts and slopes when the two detergents were used at 60°C, but very different intercepts and slopes when the two detergents had been used at 30°C. This observation was supported when slope analysis (Ho: $\beta_1=\beta_2=\beta_3=\beta_4$) confirmed significant differences between HDL at the two temperatures ($F=12.85$, d.f.=1,8, $p \leq 0.01$), between PHOS at the two temperatures ($F=31.88$, d.f.=1,8, $p \leq 0.01$) and, yet, no significant differences between the two detergents at 30°C ($F=3.76$, d.f.=6,8) or at 60°C ($F=0.02$, d.f.=1,8). The significant differences among the models were due in part to differences in intercept at the *y* axis, (Ho: $\alpha_1=\alpha_2=\alpha=_3\alpha_4$) ($F=15.46$, d.f.=3,8, $p \leq 0.01$) indicating observable differences at the two temperature levels. An important phenomenon observed in these models at the lower washing temperature was for increasing concentration of detergent to hinder residue removal when HDL detergent had been used while increasing concentrations of detergent were important to assisting in residue removal when the PHOS detergent had been used. For the PHOS detergent, as thermal energy was decreased, additional chemical energy in the form of increased measure of detergent concentration was helpful. Water hardness may have been a contributing, although not a statistically significant factor. For the HDL detergent, increased concentration of product may have decreased mechanical energy. Clearly, for the HDL, increasing detergent concentration over one and one-half times of the manufacturers recommended amount is not helpful, and under certain circumstances, may be detrimental to residue removal.

The percentage of residue remaining after laundering is a way to study the data in normalized units, given that the initial contamination of the UN specimens was twice that of the SR finished specimens. Residue retention on UN specimens was one-tenth of initial contamination; while residue retention on SR specimens was one-third of contamination.

The percentage of residue remaining on SR specimens ranged from 26.5% (HDL with prewash product) to 46.1% (PHOS without prewash product), reflecting the difficulty in removing pesticide from SR finished fabric. A GLM ANOVA was used to test for the main effects of fabric finish, detergent type, detergent concentration, prewash product use and water hardness on the percent residue remaining after laundering and all interaction effects of these factors.

The residue retained after laundering (expressed as percentage of initial contamination) was significantly influenced by fabric finish, temperature and use of prewash product. The interaction of various factors is important, for example, the use of the repellent finish had a marked impact on residue retention, and percent residue varied with detergent type, detergent concentration, and use of prewash product. Use of prewash product enhanced residue removal as compared to not using prewash product for the SR specimens, but the impact of use of prewash product was not as great for UN specimens. In fact, when the comparisons were made between the results of ANOVA for amount of residue remaining and ANOVA for percent of residue

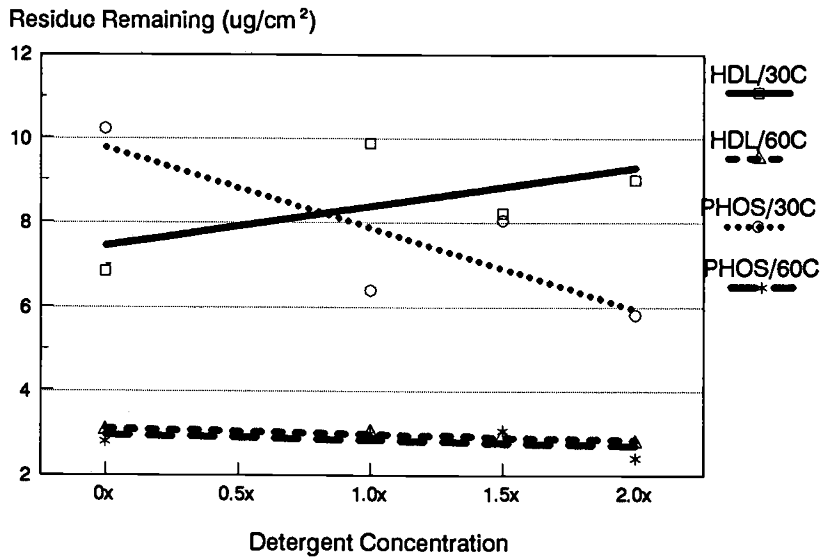

FIG. 4--Interaction of detergent type with temperature with detergent concentration on amount of residue remaining on specimens after laundering.

remaining, the impact of fabric finish was readily apparent, with interactions of temperature and finish, of prewash product and finish, and of prewash product and hardness, all which had not been found when amount ($\mu g/cm^2$) of after-laundering residues were considered.

When the interaction of detergent and finish was considered, LS Means test revealed that the difference was attributable to the finish, with similar responses between the detergent types for the UN (HDL=8.7%, PHOS = 9.9%) and SR (HDL=35.8%, PHOS = 30.3%). When the interaction of temperature and finish was studied, the response for UN washed at 60°C was clearly superior (3.3%), with the UN at 30°C (15.7%) and the SR at 60°C (18.9%) similar in response, and the highest percent of residues for the SR washed at 30°C (47.6%). Use of prewash on UN resulted in lowest residues (5.5%) while SR washed without prewash product resulted in the greatest proportion of residue remaining on the specimen after laundering (40%). Use of prewash product was important, with no significant difference at the three levels of hardness when it had been used (17.8, 15.8 and 15.8% at 0, 1,000 and 2,000 ppm, respectively) and significant differences when it had not been used (21.0, 27.6, and 29.4% at 0, 1000 and 2000 ppm, respectively). Orthogonal contrasts on these hardness data revealed a linear relationship; as water hardness increased, residue retained increased (F=9.01).

Contributing to these two-way interactions was the interaction of detergent type/temperature/ prewash and finish (Figure 5) and thus caution should be exercised about interpreting these two-way interactions without consideration of the other interaction. Although trends were similar between the amount of residue retained after laundering (Figure 3) and the percentages retained (Figure 5), greater amounts remained on the SR fabrics and given the smaller

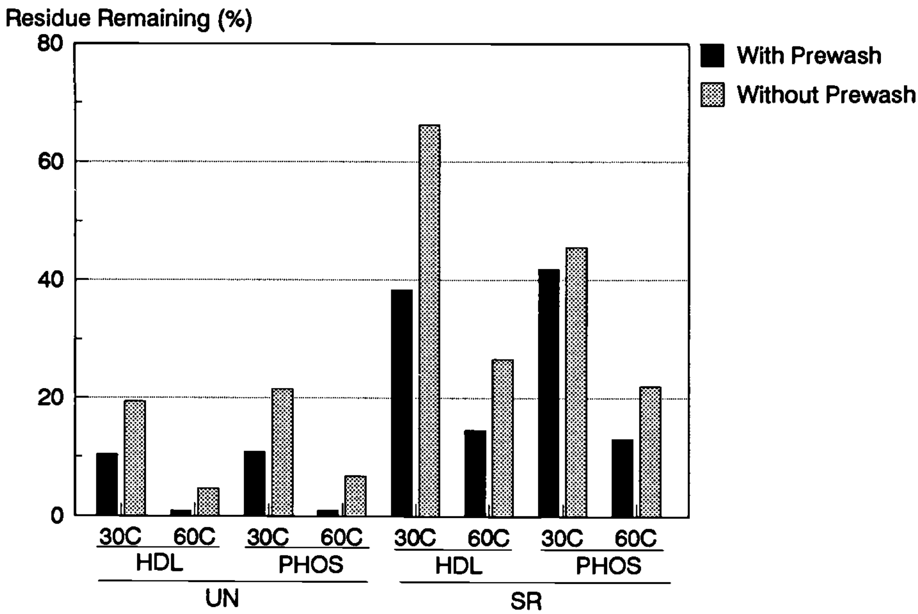

FIG. 5--Interaction of detergent type, temperature, prewash product use and fabric finish on percent of MeP remaining after laundering.

initial contamination, the percentages were much larger for the SR than for the UN. This is quite visibly discernable when comparing Figure 3 and Figure 5. Thus, the impact of the lower wash temperature, especially without the use of prewash product, on the SR finished fabrics resulted in very large residues remaining after laundering as a proportion of initial contamination, reflecting the problem with soil removal from a repellent finished fabric. Based on these findings, recommendations are made to vigorously treat SR fabrics in laundering, particularly to include a prewash product and the hottest temperature possible, regardless of laundry detergent used. As level of water hardness increases, the use of prewash product and hottest temperature possible are important if a phosphate powdered detergent is the product of choice.

Further contributing to these interactions was the significant detergent type x detergent concentration x fabric finish interaction. Model analysis was used to test the comparability of successive levels of detergent concentration for each detergent type/fabric finish combination on the percentage of residue retained by specimens as a proportion of initial contamination. Using SAS Regression analysis, the four models, represented in Figure 6, were determined to be different models ($F=11.40$, $df=6,8$, $p=0.001$). Visual inspection resulted in hypothesizing that similarities existed in the models for the UN specimens and similarities existed in the models for the SR specimens, but that the placement on the *y* axis predicted different intercepts. Slope analysis (Ho: $\beta_1=\beta_2=\beta_3=\beta_4$) failed to confirm this, with a decision to fail to reject the null hypothesis (F=1.12, d.f.=3,8, p=0.39); therefore, it was concluded that the four models were identical in slope. Intercept analyses (Ho: $\alpha_1=\alpha_2=\alpha_3=\alpha_4$) confirmed that the difference in the four models was attributable to the placement on the *y* axis (F=4.37, d.f.=3,8, p=0.04), with observable differences between the two fabric finishes. This finding does confirm the previous observations for more effective residue removal from unfinished fabrics and the concomitant conclusion to vigorously treat repellent finished fabrics during laundering.

IMPLICATIONS

Methyl parathion residues are more effectively removed from unfinished cotton/polyester fabric than they are from repellent finished fabric. The results of this study indicate soil repellent finished fabrics warrant more vigorous laundering treatment to lower residues retained in fabric. Increasing thermal energy or achieving a different combination of chemical energies will assist in residue removal. Use of prewash product enhances residue removal, and especially if the detergent selected for laundering is an anionic phosphate detergent, and is used in areas of hard water. In areas with hard water, water softening products or ion exchange equipment to soften water may be an alternative. Using a prewash product and/or increasing the detergent concentration, especially at lower temperatures of washing, is recommended.

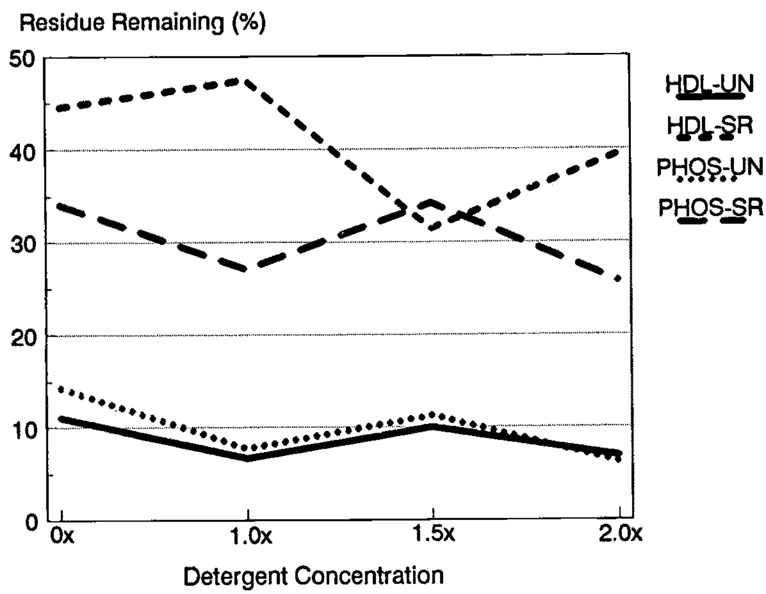

FIG. 6--Percentage of residue remaining on UN and SR fabrics attributable to detergent type and detergent concentration.

Even though pesticide removal is more of a problem from SR finished fabric than from an UN fabric, the fact that the repellent finish limits contamination levels supports the recommendations [1, 5, 7, 12] that SR finish be used on applicator work garments. Increasing the detergent concentration is much more important when laundering is done without the use of a prewash product. Based on these data, the recommendation that elevated wash temperatures and a prewash product be used along with increased detergent in laundering pesticide-contaminated clothing, especially for a repellent finished fabric, is appropriate.

Acknowledgments

This research was supported in part by the Nebraska Agricultural Research Division Project 94-015 and contributes to North Central Research Project NC-170, "Reducing Pesticide Exposure of Applicators through Improved Clothing Design and Care." This paper is published as Journal Series Number 9186, Nebraska Agricultural Research Division. The dedicated assistance of Kristina Newburn and Wendelin Rich is appreciated. The dedicated assistance of Kristina Newburn and Wendelin Rich in the laboratory and Alex Polymenopoulos, as statistical consultant is acknowledged.

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